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GROWTH OF SAPPHIRE DISKS FROM THE MELT BY A GRADIENT FURNACE TECHNIQUE

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CERAMICS DIVISION

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Technical Report by

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ABSTRACT

Single crystal Al_2O_3 (sapphire) is a candidate material for various window applications. A gradient furnace technique has been developed for the growth of large sapphire disks, 7.3 cm in diameter and 1.5 cm thick, from the melt in molybdenum crucibles. The disks are free of gas bubbles and inclusions and consist of several large pie-shaped grains. Within the grains dislocation densities of 1×10^5 cm^{-2} have been measured. The disks are essentially transparent to radiation from wavelengths of 0.25 μm in the ultraviolet spectrum to 4.5 μm in the infrared spectrum.

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GROWTH OF SAPPHIRE DISKS FROM THE MELT BY A GRADIENT FURNACE TECHNIQUE

Single crystal Al_2O_3 (sapphire) is a candidate material for various window applications in the visible spectrum and in the infrared spectrum. Growth of sapphire from the melt as reported in the literature has been accomplished using a Verneuil technique,[1] a Czochralski technique,[2-4] and a floating zone technique.[3,4] These techniques, however, did not appear to offer the flexibility required to fabricate large single crystals in significant quantities for various window applications. New crystal growth techniques from the melt would have to be developed if sapphire were to be widely used as a window material. It was felt that a casting or controlled solidification technique, where the melt was contained in a crucible and growth of the solid circumscribed by the crucible, could be developed. A gradient furnace technique was evolved for the growth of sapphire disks. This report will describe the technique and the sapphire disks produced.

The gradient furnace is a vacuum graphite resistance furnace* with a helium-cooled tungsten/molybdenum heat exchanger projecting up into it as illustrated schematically in Figure 1. The material to be melted, in this case previously fused Al_2O_3, is contained in a molybdenum crucible which is seated atop the heat exchanger. A seed crystal is located at the base of the crucible in the center directly above the heat exchanger. The material is melted except for the seed

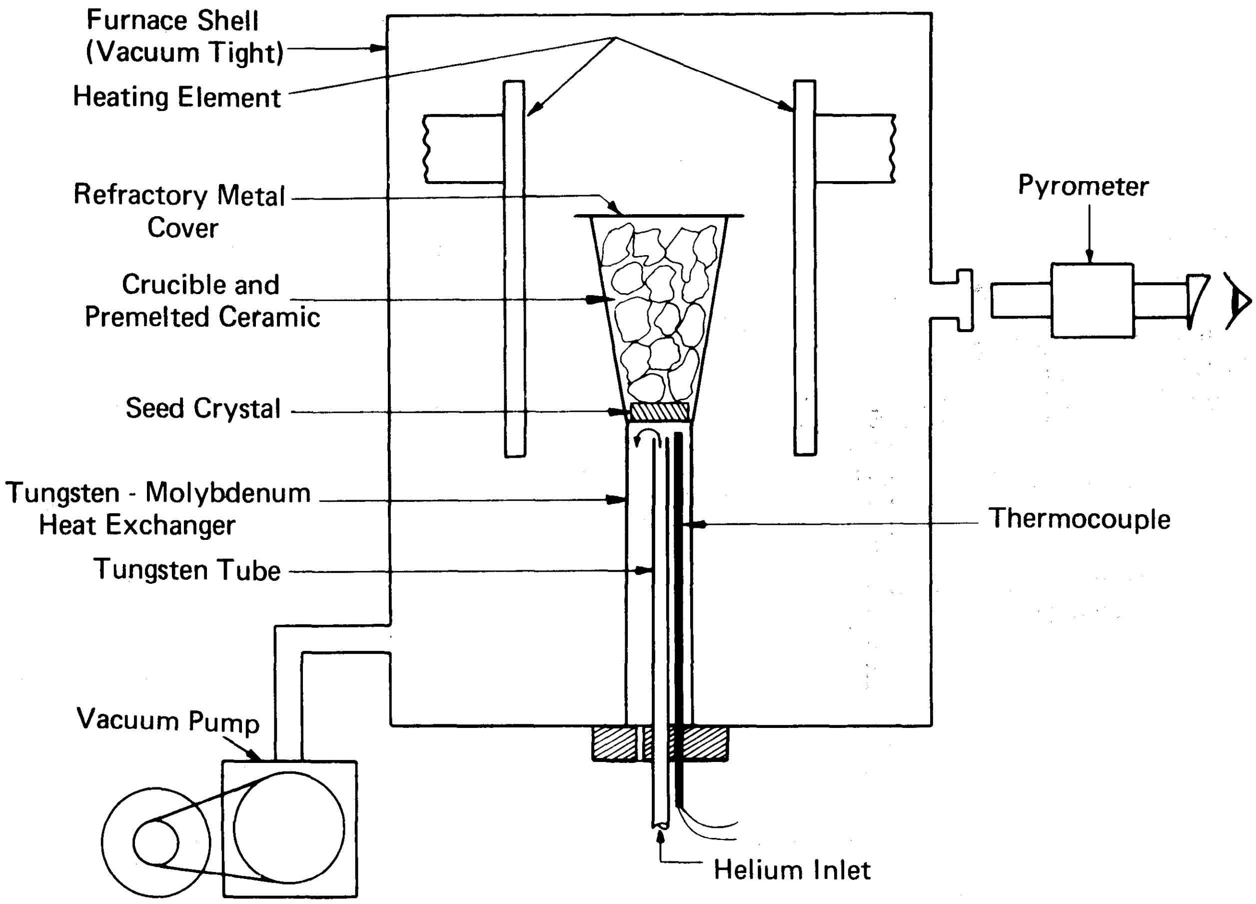

Figure 1. SCHEMATIC OF GRADIENT FURNACE

*Vacuum Specialties, Inc., 34 Linden St. Somerville, Massachusetts

crystal which is cooled by the heat exchanger directly below it. The helium flow through the heat exchanger is increased and more heat is removed from the material. The melt nucleates on the seed crystal, and the solid grows out from the seed crystal. The driving force for growth is created by the temperature gradients set up in the material as the heat flows from the heating elements, through the material, and out the heat exchanger where the bulk of it is removed by the helium. The technique is flexible in that a variety of crucible shapes and sizes may be used. A conical crucible is illustrated in Figure 1. Figure 2 illustrates the arrangement used to grow sapphire disks where a flat dish-shaped crucible is employed. Two sapphire disks are seen in Figure 3. The one on the left, 7.3 cm in diameter and 1.5 cm thick, is just as removed from the crucible with no machining. The one on the right, 7.3 cm in diameter and 0.9 cm, has been ground and polished to a submicron finish* and a portion removed for other studies. Semi-quantitative spectrochemical analysis† of similar disks revealed that molybdenum content is below 100 ppm indicating that reaction between molten Al_2O_3 and the molybdenum crucible is minimal. No gas bubbles were observed with the naked eye or with a petrographic microscope to 500X. When the disks were observed in a polariscope, a structure consisting of wedge-shaped columnar sub-grains radiating from the center of the disk was seen. X-ray analysis of the disks was conducted using the Laue method. Laue patterns obtained at various locations on both sides of the disks revealed that the grains were misoriented by less than 10°. The disks in Figure 3 are oriented so that the $(11\bar{2}0)$ planes are nearly parallel to the faces of the disks. Dislocation densities on the (0001) plane were determined in the interior of the large grains after etching with hot orthophosphoric acid.[5] Dislocation densities of 1×10^5 cm^{-2} were obtained which were comparable to sapphire grown by the Czochralski technique.[3-5] Absorption edges of a sapphire disk 0.9 cm thick were determined

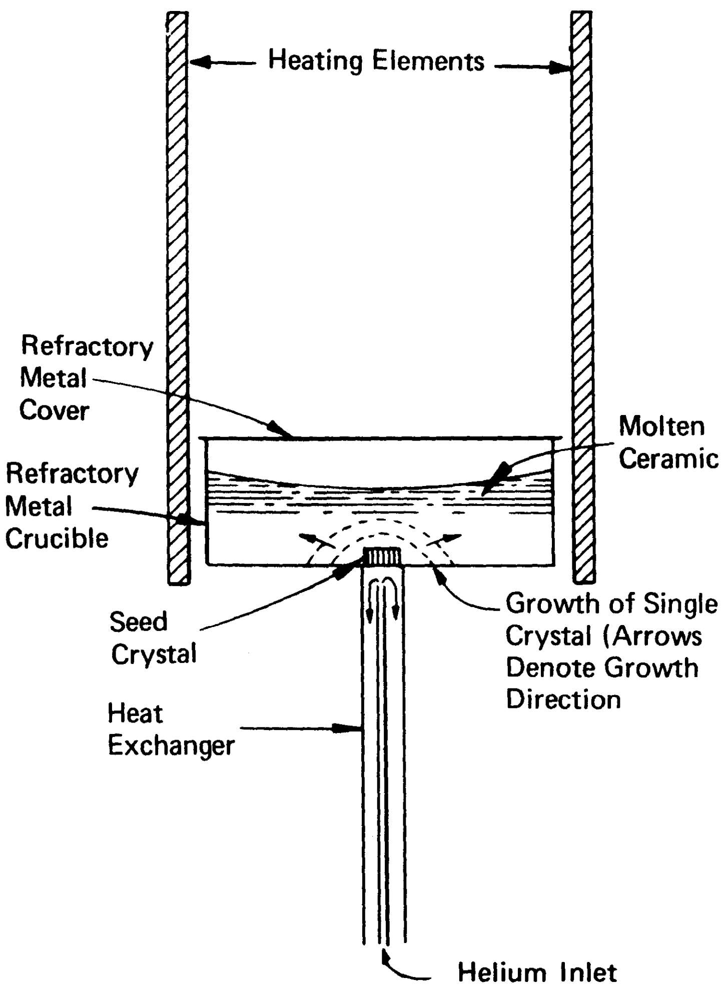

Figure 2. SCHEMATIC ARRANGEMENT IN THE GRADIENT FURNACE FOR THE GROWTH OF SAPPHIRE DISKS

*Bomas Machining Specialties, Inc., 114 N. Beacon St., Brighton, Massachusetts

†Jarrel-Ash Division of Fisher Scientific Company, Waltham, Massachusetts

using three spectrophotometers* and the results are shown in Figure 4. The disk was essentially transparent to radiation from 0.25 μm in the ultraviolet to 4.5 μm in the infrared.

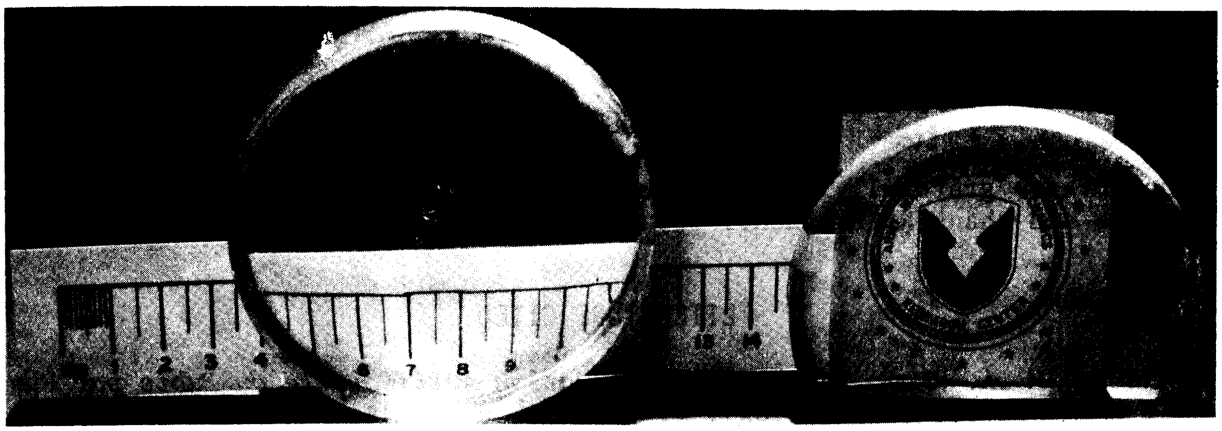

Figure 3. SAPPHIRE DISKS GROWN BY THE GRADIENT FURNACE TECHNIQUE. The one at the left is as removed from the crucible with no machining. The one at the right has been ground and polished to a submicron finish and a portion was removed for other studies.

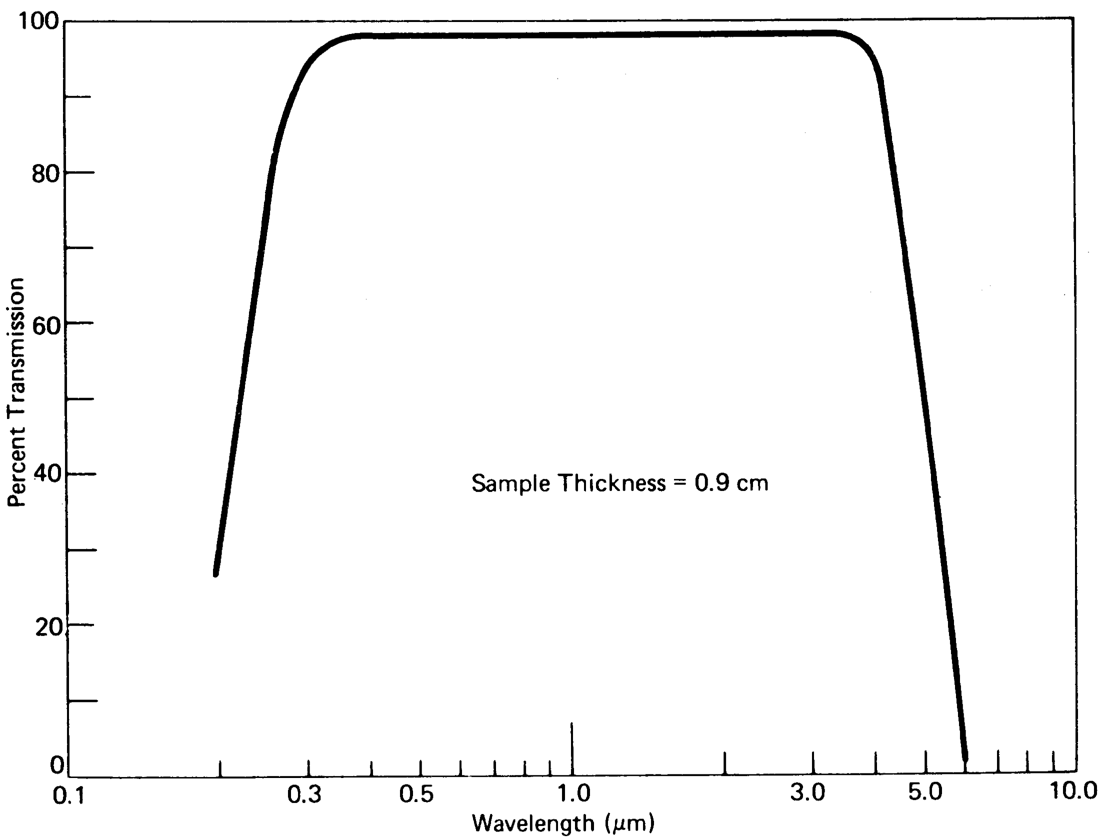

Figure 4. A PLOT OF PERCENT TRANSMISSION VERSUS WAVELENGTH FOR SAPPHIRE GROWN BY THE GRADIENT FURNACE TECHNIQUE

*Model 202 Ultraviolet-Visible Spectrophotometer, Perkin-Elmer Corp., Main Avenue, Norwalk, Connecticut; Models IR-12 and DK-1A Spectrophotometers, Beckman Instruments, Inc., 2500 Harbor Blvd., Fullerton, California

A casting or controlled solidification technique employing a gradient furnace has been developed for the growth of large sapphire disks. Sapphire disks 7.3 cm in diameter and 1.5 thick have been reproducibly grown to shape in molybdenum crucibles. The simplicity and flexibility of the technique makes it attractive for growth of even larger crystals required for lightweight, transparent armor applications.

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